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Recovery of Plutonium-244 from the Mark-18A Targets at Savannah River Site

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EXECUTIVE SUMMARY

In recent years, the Department of Energy – National Nuclear Security Administration (DOE-NNSA) Office of Nuclear Material Integration (NA-532) has tasked Savannah River Site (SRS)/Savannah River National Laboratory (SRNL) to develop a process to recover plutonium from the Mark-18A (MK-18A) targets. The Mk-18A plutonium isotopes are unique, and contain the world's remaining, unseparated supply of plutonium-244. The current accessible inventory of plutonium-244 was recovered in the early 1970's at Oak Ridge National Laboratory (ORNL) from twenty-one Mk-18A targets. The demand for plutonium-244 is growing beyond the current inventory as increasing demands for nuclear proliferation monitoring and mixed oxide fuel production all benefit from the use of plutonium-244 in analytical analysis.

Today, sixty-five Mk-18A targets are currently stored in SRS's L-Basin. A large, multidisciplinary team has been established at SRNL (with additional support for ORNL staff) to develop the method to recover the valuable plutonium at SRNL and ship the radiological material to ORNL. The targets will be removed one at a time for transportation from L-Basin to SRNL in a newly designed onsite transportation cask. Once received into SRNL, the targets will be loaded into SRNL's shielded cell facility, where they will be processed to recover the plutonium for future isotopic purification at ORNL. The remaining high burn-up actinides (higher isotopic distribution of Am, Cm and Cf) will also be sent to ORNL for future programs.

During the development of the process flowsheet at SRNL, the main focus is to ensure its practicality for operation with maximum recovery of plutonium-244. Additional considerations in the design are meeting the current facility authorization basis, controlling potential off-site releases in unmitigated accident scenarios, minimizing worker exposure and meeting a purity specification for shipment of radiological materials to ORNL. At this time, the first Mk-18A target is expected to be received in SRNL late-2020.

INTRODUCTION

Mark-18A is the Savannah River Site (SRS) designation for the special target assembly irradiated in K-reactor at SRS from August 1969 until June 1979. Mark-18A targets are unique not only in the duration of irradiation but in that for the first fifteen months of irradiation, the targets were subject to neutron fluxes reaching 1.8×10^{15} $\eta/\text{cm}^2 \cdot \text{s}$. That extremely high neutron flux allowed the starting material comprising ~98% plutonium-242 to produce gram quantities of plutonium-244. (The extremely short half-life of plutonium-243 essentially prevents the neutron capture power reactors.)

The original loading of K-reactor was eighty-six Mark-18A targets, of which twenty-one targets were removed from the reactor and shipped to the Radiochemical Engineering Development Center (REDC) at Oak Ridge National Laboratory (ORNL) during the early 1970s.¹ REDC recovered the californium, curium which had a significant amount of 246 & 248), and plutonium. The plutonium was later isotopically purified to create high assay plutonium-244 material, which has been used in a wide range of programs and mass spectroscopy standards.

At the removal of the remaining sixty-five Mark-18A targets from K-reactor in June 18, 1979, it was noted that the exterior cladding had significant surface oxidation; some also had blistering and observable pit corrosion. Because when the targets would be processed and whether the cladding would maintain structural integrity in the long term were unknown, the Mark-18A targets were resized (by removal of aluminum tubing used to center the target in the reactor core) and over-packed into “J-cans,” each consisting of two concentric vented tubes. The total height of the outer J-can is fourteen feet, and the outer diameter is 5.563”. The Mark-18A targets in the J-cans have been continuously stored underwater at SRS, currently in L-basin.

In recent years, the Office of Nuclear Material Integration within the Department of Energy’s National Nuclear Security Administration (NA-532) has tasked SRS, Savannah River National Laboratory (SRNL) and ORNL to develop the process needed to recover the plutonium from the Mark-18A targets and then ship the irreplaceable plutonium to ORNL.

In order to recover the plutonium-244, the Mark-18A targets have to be separated from the J-can and the aluminum cladding removed from the plutonium (which is mixed with transplutonium and fission products). The high radiological dose (both gamma from the fission product and neutron from the transamericium elements) will require the Mark-18A to be processed in an appropriately shielded facility. The remaining sixty-five Mark-18A targets contain about 400 g of mixed isotope plutonium (with <25 g plutonium-244), thus highly efficient separation of the plutonium, without cross-contamination, is highly desired.

SRNL led the evaluation of all facilities at SRS in which the plutonium from the Mark-18A could potentially be recovered. Based on facility capabilities and funding profiles for all the options, the shielded cells within SRNL were selected as the best location to recover the plutonium from the Mark-18A. In order to recover the plutonium, SRNL is going to have to design, build, and install a significant amount of new equipment into the shielded cells. The entire process and controls have to be bounded within the documented safety analysis of the facility, as no new credited infrastructure will be built for this program. Additional constraints of the project include the absence of a Department of Transportation (DOT) approved shipping container for the Mk-18A and the inability of REDC to handle the 14’ height of the J-can.

DEVELOPMENT OF FLOW SHEET FOR RECOVER OF MARK-18A

Define the Source Term²

Even though the Mark-18A targets were built, irradiated, and stored on site, an accurate source term of the isotopic distribution did not exist. During the irradiation of the targets, only the actinide build-up was calculated (i.e., not the fission products), and only five fission isotopes were reported in site reports. The software that calculated the actinide growth was a site-created reactor code, optimized for the production of weapons-usable plutonium from a thermal neutron capture of uranium-238. This reactor model calculated in the late 1970s was inadequate for today's processing of the Mark-18A. Mark-18A fissions are mostly of transplutonium elements, which cause a shift in the fission distribution profile.

All work conducted at SRNL will be based on a new source term, which will be used for shielding requirements for on- and off-site transportation, potential worker exposure, flow sheet development, material-at-risk within the facility, etc.; thus the source term has to be defensible to external reviewers. SRNL turned to computational reactor modeling code using modern isotope cross sections and computational capabilities that were not available in the 1970s. The model did have some conservative assumptions, which were selected to bind the radiological dose (specifically the neutron dose).

The reactor model was separated into two distinct time frames of irradiation as it allowed for easier computer modeling to handle the changes in the reactor configuration. In the model, all eight-six targets were loaded with 120 g of plutonium (98% assay plutonium-242) oxide. No variation in the z-direction neutron flux profile, as the Mark-18A target region is only four feet, and the drivers were nominally six feet of highly enriched uranium.

During the first fifteen months of irradiation, K-reactor was dedicated to the irradiation of the Mark-18A. With no other campaigns during this time, the active area of the reactor was decreased, allowing a higher coolant flow rate to be achieved in the active channels and higher flux rate to be achieved. The Mark-18A received 88% of their total fluence, $9.23 \times 10^{22} \text{ } \eta/\text{cm}^2$, with an average flux rate of $1.8 \times 10^{15} \text{ } \eta/\text{cm}^2 \cdot \text{s}$ during the first fifteen months. The initial core composition was molded with TRITON and was broken down into three concentric circles for reactor region bins (to account for the difference in neurotics due to the compressed reactor core). The absorber concentration was varied until the model achieved a k_{eff} of 1.0 and the neutron flux shape was calculated with KENO for the three reactor region bins. For this reason, reported peaking factors for the run were used, rather than the model values, since the absorber concentrations were not constant in reality. No resonance self-shielding (RSS) was used, as the targets are extremely thin and low density. The specific cross section libraries were created using T-DEPL module from the specific reactor and target geometry and compositions. Depletion of the drivers was performed by ORIGEN-S and used the daily thermal power to calculate the flux, using the average thermal cross section calculated in T-DEPL. The ADNUX 4 optional parameter was used to ensure the inclusion of the plutonium-243 cross section in the Mark-18A targets (which is omitted by default in SCALE).

The final eight and half years were modeled in MCNP5, and all targets were modeled in the most conservative (highest curium production) location for bounding neutron dose calculation. The targets were then cooled to January 1, 2106. Each target has just less than 500 Ci of activity, based on the initial loading of 120 g plutonium oxide.

Caustic Dissolution of Aluminum Cladding³

After loading the Mark-18A and J-can into SRNL's shielded cell facility the first task is to removal the Mark-18A target of the J-cans. The shielded cells facility is only six feet deep and the outer J-can is fourteen feet long. Each Mark-18A target has about twelve and a half pounds of aluminum cladding that has no value and thus will be dispositioned at SRNL. In view the entire flow sheet, it was decided that having two discrete dissolution steps would provide a significant decontamination factor (D_f) for aluminum and silicon (mostly activation product of the aluminum cladding). The aluminum cladding will be dissolved via caustic dissolution and the target material via nitric acid, discussed later.

Caustic dissolution of aluminum was used in SRS's processing canyons for decades, and REDC used caustic dissolution to remove the cladding from the twenty-one Mark-18A targets that were processed in the early 70s. From the REDC records, there was carry over of aluminum into the acid dissolution phase. It was assumed to be due to surface oxidized aluminum (Al_2O_3), which does not dissolve in caustic. Even with this small amount of carry over, the caustic dissolution of aluminum will provide a significant removal of aluminum.

Caustic dissolution is kinetically slow at temperature below $80^\circ C$, but is an extremely exothermic reaction ($\sim 110 MJ/Mark-18A$). The reaction also releases over 1.4 pounds of hydrogen, if not suppressed with the addition of nitrate (NO_3^-). The suppression of hydrogen is highly favorable as it shifts the off-gas products to the less flammable ammonium, reaction 1.



Due to SRNL's documented safety analysis, the off-gas rate within the shielded cell facility has to stay below 25% of the lower flammable limit (LFL). Thus the addition of excess nitrate is beneficial by allowing for faster dissolution of the aluminum cladding, while maintain less than the 25% LFL (sum of H_2 and NH_3).

Through a detailed test matrix varying initial volume concentration of caustic and nitrate, temperature profile of the dissolution, and the concentration and addition rate of caustic to replace consumed caustic, SRNL has proposed a flow sheet baseline for a quarter of a Mark-18A target.

The decision for SRNL to dissolve a quarter of a Mark-18A target at a time is to help support further downstream operations and maintain less than the 25% LFL for flammable gas generation within the shielded cells. Working with slightly small amounts of material should allow for a semi-batch operation, smaller equipment footprint, higher efficiency, and faster turnover on SRNL's standard work schedule.

The dissolver will be designed in out years and will have several key features to address temperature control and off-gas. Since caustic dissolution is kinetically slow at temperatures less than $80^\circ C$ the dissolver will have to be initially heated to $80^\circ C$. Once the dissolution has started, the exothermic reaction could cause an increase in the solution temperature and thus increase the reaction rate. There are two ways to control the reaction kinetics, temperature and available caustic that will be designed into the system as the LFL within the shielded cells must be maintained.

The dissolver design will also have to incorporate the loading the target into the container, removing of liquid and solids, and the off-gas releases. All of these parts are important due to the potential ways to control contamination within SRNL's shielded cell facility and interfacing with the manipulator and in-cell gantry crane.

SRNL's current baseline caustic dissolution will start with an initial solution of 2.1 M NaNO_3 /0.425 M NaOH under an inert purge gas (e.g., N_2). A quarter of a Mark-18A target will be loaded into the dissolver, and then the dissolver solution will be externally heated to 80°C. An 80 °C operating temperature will be held for two hours before heating the solution to 85 °C for an hour and a half. The remaining process cycle, the solution will be held at 90 °C (predicted to take an additional two and half to three additional hours). This will allow for the entire caustic dissolution to be completed within one normal operational shift.

Filtration of Caustic Solution ⁴

As the majority of the Mark-18A target material (fission products and actinides) will not be soluble in the caustic solution, the slurry will have to be filtered after the cladding is dissolved. The filtering will provide the initial separation of the actinides from the aluminum and silicon. Though it is expected that there will be some small amounts of loss due to actinide solubility in caustic, the amounts should meet the current waste criteria for disposition.

Filtration of the caustic solution is important due to the time dependent ingrowth of gibbsite $\{\text{Al}(\text{OH})_3\}$, which is highly hydrated and has poor filtering properties. (Gibbsite will be used generically for the aluminum hydroxide solid; in our studies most was in the bayerite crystal structure.) The growth of gibbsite is kinetically slow, and the solubility is very sensitive to temperature. These two factors encourage maintaining the solution at an elevated temperature and filtering as soon as possible.

The transmutation of aluminum into silicon is assumed to be negligible and thus does not complicate the processing of aluminum cladding. In the case of the Mark-18A, the fluence was significant to transmute almost 3% of the aluminum to silicon. This additional silicon could complicate the filtering processes. It was not observed in the experimental work conducted for the Mark-18A, but thermodynamic calculations indicated that sodium aluminosilicate would also precipitate in the caustic solutions.

The other potential variable the SRNL considered is the filter media average pore size. Small testing evaluated a range of stainless steel filters media (grades 2-20) over potential operations temperature profile. The test matrix allowed for evaluation of the initial permeance (as a filter cake is collected the permeance decreases) and the effects of temperature on permeance.

In the future design of the caustic filtering system, a standard sintered stainless steel filter pore size of media grade 7 or greater will provide the best permeances. Any decanting abilities from the caustic dissolver will significantly decrease the filtering time. Additional, the caustic solution, transfer lines and the filter housing will be maintained at 60 °C. At this temperature the permeance is sufficiently increases to justify the additional design requirements.

Acid Dissolution of Mark-18A Target Material and Oxidation Adjustment ⁵

After the insoluble solids are removed from the sintered stainless steel filter, media will need to be dissolved for downstream purification. Traditionally, plutonium oxide bearing material is dissolved in strong nitric acid with the addition of fluoride (typically as HF). Though the fluoride is somewhat corrosive to glass, stainless steel does not demonstrate significant corrosion. REDC's material of construction includes Zircaloy-2 and tantalum which are more susceptible to fluoride corrosion. It has been requested that SRNL not dissolve the material with the addition of fluoride, if possible.

This unit operation is complicated by the fact there is no appropriate surrogate. What would the surrogate be? How is the dissolution of plutonium oxide with significant amounts of incorporated fission product differ from pure plutonium oxide? How does the ten years of irradiation affect the materials structure matrix? The almost forty years of storages has allowed 2.2 half-lives of curium-244 to decay into plutonium-240, what is the effect on the kinetics of dissolution?

Due to the lack of a surrogate, SRNL based this unit operation on literature review, and scientific knowledge of sixty years of history. Literature demonstrates that irradiation of plutonium oxide 'breaks-up' the crystalline lattice enough to significantly facilitate the dissolution without the addition of fluoride. ORNL was able to provide documentation of the early 1970's recover of the Mark-18A at REDC and they had no difficulties in dissolving the Mark-18A target material in just nitric acid. Though no mass balance information was collected, and no comments addressing if any undissolved solids (UDS) were present. SRNL expect there will be a significant amount of UDS due to the fission product profile.

The longer SRNL has to heat the Mark-18A material in nitric acid to get it to dissolve, the amount of plutonium (VI) will increase even in 7-8 M HNO_3 . The presence of plutonium (VI) will decrease the effective recover in the anion exchange step, unless the system reduced back to plutonium (IV). This is another reason why dissolving of the Mark-18A as quickly as possible is beneficial to the program as a whole.

SRNL will be prepared for the material that did not dissolve in just nitric acid, by the means of a peroxide fusion. Even if the peroxide fusion is not needed for the dissolution of plutonium, it will be used for the dissolution of the UDS that will be collected over several Mark-18A targets. The processing of the UDS will ensure that all the plutonium will have a chance to be separated and facilitate disposition of the fission products at SRNL.

No matter the method of dissolution, it is expected that there will be some plutonium (VI) that will need to be reduced to plutonium (IV) prior to moving to the next unit operation of anion exchange. Initial work into oxidation controls evaluated the use of nitric oxide (NO) and nitrogen dioxide (NO_2). The use of a gaseous species was preferred for waste minimization and product purity. Neither reductant in the presence of non-radiological fission products was able to reduce plutonium (VI) to plutonium (IV).

Current testing and flow sheet development is underway to validate that ferrous sulfamate will reduce any plutonium (VI) that might be produced during dissolution. Ferrous sulfamate will reduce plutonium to plutonium (III) and thus will require oxidation to plutonium (IV) with the addition of nitrite (NO_2^-). Based on the assumption that ferrous sulfamate/nitrite will work, the baseline flow sheet will be the first Mark-18A's plutonium will be reduced with ferrous sulfamate then oxidized with nitrite prior to loading the anion exchange resin. After eluting the anion exchange, this clean plutonium will then be held within SRNL's shielded cells facility until the next Mark-18A is received into the facility. The previous Mark-18A plutonium will have the same isotopics distribution and be chemically clean, and thus can be reduced (e.g., ascorbic acid) to create a plutonium (III) stock without diluting the plutonium-244. This plutonium (III) stock will be used as the reducing agent in the Mark-18A program.

*Anion Exchange of Mark-18A Target Material for the Separation of Plutonium*⁶

There are several different ways to separate/purify plutonium; it was the decision of SRNL to approach the purification through anion exchange. The bases of this approach over the more

traditional solvent extraction (e.g., PUREX) is simplicity of equipment, reduction of liquid waste, no liquid organic waste, ease of operation within the shielded cells facility and SRNL extensive history in anion technology.

Recently, SRNL validated the use of anion exchange for SRS's HB-Line purification of aged plutonium for potential mixed oxide (MOX) feed. This program used the Reillex-HPQ anion exchange resin, and thus the Mark-18A program is going to focus on the same resin.

The Mark-18A program could not solely rely on the previous research for the HB-Line program because that program's feed stock was high assay plutonium with lesser amounts of transition metal impurities and no curium. Also control of the oxidation state of plutonium was less of a problem as higher plutonium losses were acceptable for that mission. For the Mark-18A the purity requirement for the plutonium is less (for the work completed in the shielded cells) as the plutonium will be transferred to a radiological glovebox for additional purification prior to shipping.

From the dissolution of the Mark-18A, there will be a significant amount of transition metals, lanthanide and transplutonium elements in the matrix. Though anion exchange has been used for decades because of its high efficiency in separation, it is not always well documented. In order for SRNL to optimize the separation, a better measurement of the D_f for each group of elements is needed, with focus on the lanthanides (III) and high gamma emitting isotopes. Since SRNL does not have a curium stock to validate its D_f , the lanthanide series will have to be used along with the measured D_f for americium to predict the needed operational parameters for purification.

Other parallel work has focused on predicting the anion's resilience to the expected radiological dose and chemical exposure. Through computational modeling the resin's storage under nitric acid causes more degradation than the curium-244 alpha exposure. Scheduled replacement of the resin will be part of the operational plan, once in the shielded cells.

One of the other issues for the longevity for the anion exchange is the high affinity for palladium and its low stripping efficiency. Due to the Mark-18A having a shifted fission profile, more palladium was produced than traditional thermal fission of uranium thus will affect the resin capacity. Recover of the palladium has no priority at this time, so disposition to waste is the current path forward.

*Concentrating, Denitration, and Oxalate Precipitation of Fission Products and Transplutonium Elements*⁷

It is currently estimated that after the loading and wash solution from the anion exchange operations is collected, each quarter of a Mark-18A target will produce between five and eight liters of nitric acid. This solution will contain the majority of the fission products transplutonium isotopes from the Mark-18A, which have no future mission at SRS. With the exception of plutonium and noble gases, the fission products include most of the cesium and palladium and some of the strontium and other mildly caustic soluble elements, and the transplutonium isotopes include ²⁴¹⁻²⁴³Am, ²⁴⁴⁻²⁴⁸Cm and ²⁴⁹⁻²⁵²Cf. The current material path for the transplutonium elements is shipping to the ORNL REDC for future feed stock in other campaigns/programs.

Shipping the transplutonium rich material to ORNL will be expensive and a potential for high radiological exposure to the SRNL and ORNL workers. The goal is to ship as few shipping containers with highest assay of transplutonium elements per container. The material will be package into a certified special forms capsule (Los Alamos National Laboratory designed SFC

Model III). The use of a certified special forms capsule will allow SRNL to use a Type A package, which provides significantly more shielding options than a Type B package. Disposition of the remaining fission products will occur at SRNL as they fit within our normal operating parameters and designed facilities.

The large amount of unwanted nitric acid and fission products that SRNL can easily disposition and the desire to ship the transplutonium elements to ORNL are the driving forces to provide one additional purification step. SRNL's baseline study is a distillation, denitration, oxalate precipitation, and then calcination methodology.

The feed acid will have a low concentration of elements in a moderate acid concentration (~8 M HNO₃). To reduce the volume, the acid concentration will increase and approach ~68_{wt.}% HNO₃ (~14.8-15.1 M). This acid concentration (~15M) is over an order of magnitude greater than the optimal oxalate precipitation (~1 M or less), thus it has to be decreased through chemical destruction.

Once the distillation is completed, the active volume of nitric acid will be about one liter. To this nominal one liter of concentrated nitric acid and inorganics, a slow addition of formic acid will be added, at temperature. The formic acid will denitrate the solution through the formation of nitrogen dioxide (NO₂), nitric oxide (NO), carbon dioxide (CO₂), and water (H₂O). Once the nitrate concentration has reached ~1 M, the system is ideal for oxalate precipitation of the transplutonium and lanthanides elements.

Oxalate precipitation is not a new technology as it has been employed internationally for the purification of plutonium. Here, we are currently validating its use in the separation of trivalent *f*-elements from transition elements. This separation will concentrate the product into an easily calcinable material. From the open literature, small amounts of americium and curium are expected to remain in solution, but experimental results indicate changing the dielectric constant of the solution with isopropanol significantly decreases their solubility.

Packaging and Off-site Shipping of Plutonium and Transplutonium Products

The plutonium product will be shipped to ORNL via a Department of Transportation (DOT) Type B package. There is currently no Type B package approved to ship plutonium-244. This is not an oversight by the packaging regulators; it is just the consequence of the fact that gram quantity plutonium-244 does not exist except in the Mark-18A.

SRNL is going to lead the creation of a new contents envelope in the current Safety Analysis Report of Packaging (SARP) to include the required analysis of plutonium-244 in the 9975 Type B package. This amendment will also bound any other future shipments of Type B quantities of material with plutonium-244.

The trivalent *f*-element product will be packaged into a certified special forms capsule and packaged into a modified S300, a DOT-compliant Type A package. ORNL is leading this effort, and a separate presentation and paper are being presented at this conference directly related to this work.

CONCLUSIONS

Though SRNL and ORNL are still a few years away from recovering the first gram of plutonium-244 from the Mark-18A, SRNL and ORNL have used our process knowledge as well as available literature to develop the baseline flow sheet. This work could not be executed without the support of

a very large cross section of expertise within SRNL and ORNL. Each unit operation has been done to validate the baseline flow sheet for feasibility and scale-up and to provide the needed data to ensure it will work within the authorization basis of the facility. The team's approach is to reduce future risk, thus insuring that SRNL will be able to receive the first target and separate the plutonium from a Mark-18A. Future work on the flow sheet will focus on optimization to decrease processing time and/or increase the purity of the product streams.

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